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TITLE: Recovering polyvinyl-butyril from
waste - by melt blending waste with incompatible
polymer and anhydride modified polymer to form blend, used
for mfg. dustbins and lids, etc.

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PATENT-ASSIGNEE: DU PONT DE NEMOURS & CO E I[DUPO]

PRIORITY-DATA: 1991US-0734770 (July 23, 1991)

PATENT-FAMILY:

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GR IT LU MC NL SE

CITED-DOCUMENTS: EP 167455; EP 471658 ; US 5130370

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO
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1992WO-US06000	July 23, 1992	

INT-CL (IPC): C08J011/06, C08L029/14 , C08L051/06

ABSTRACTED-PUB-NO: WO 9302141A

BASIC-ABSTRACT:

Process comprises melt blending waste (I) with : (a) an incompatible polymer (II) having a melt processing temp. less than the decomposition temp. of (I); and (b) sufficient anhydride-modified polymer (III) to compatibilise the resultant blend. The blend is also claimed.

Pref. articles mfd. from the blend have a notched Izod impact strength of at least 214 J/m. Pref. the blend comprises 20-60 wt.% (I), 20-60 wt.% (II) and 2.5-20 wt.% (III). Pref. (I) is recovered from the mfr. of (I), the mfr. of glass laminates in which (I) is an interlayer between the glass sheets; and from the glass laminates after mfr. Pref. (II) is also a waste polymer and (III) is pref. succinic anhydride-modified ethylene copolymer.

USE/DVANTAGE - The blend may be used in mfr. of dustbins and lids and drain pans for motor oil. The articles have high toughness.

In an example, a mixt. of 27.5 g flakes of high density polyethylene recycle resin from soda bottle base cups, 22.0 g recovered polyvinylbutyral and 5.5 g of a copolymer of ethylene (71 wt.%)/propylene (23 wt. %)/1,4-hexanediene (6 wt.%) grafted with 1.8 wt.% succinic anhydride gps. was melt blended at 190 deg. C for 5 min. to form moulding granules. Test specimens were moulded from the granules at 190 deg. C and found to have a Notched Izod impact strength of 785 J/m. and an elongation of 290%. In comparison when the graft copolymer was omitted from the blend the notched Izod impact strength was only 21 J/m and the elongation only 74%.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: RECOVER POLYVINYL BUTYRAL WASTE MELT BLEND

WASTE INCOMPATIBLE

POLYMER ANHYDRIDE MODIFIED POLYMER FORM BLEND
MANUFACTURE DUSTBIN
LID

DERWENT-CLASS: A14

CPI-CODES: A07-A02C; A07-A04; A09-A05A; A10-E02; A10-E07;
A11-A03; A11-C03;

POLYMER-MULTIPUNCH-CODES-AND-KEY-SERIALS:

Key Serials: 0009 0038 0218 0219 0222 0226 0229 0239 0240
0242 0247 0251 1208
1992 1994 2022 2319 2332 2333 2334 2401 2403 2510 2542 2562
2585 2617 2628 2635
2667 2763 2775 2780 2781 2787 3154 3267
Multipunch Codes: 014 02& 03- 032 034 040 041 046 047 049
050 106 13& 134 231
232 233 235 250 28& 289 311 342 381 392 393 394 395 396 421
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514 531 551 556 560 566 567 57& 573 575 583 589 604 608 633
641 652 653 688 720
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(21) International Application Number: PCT/US92/06000 (22) International Filing Date: 23 July 1992 (23.07.92) (30) Priority data: 734,770 23 July 1991 (23.07.91) US (71) Applicant: E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US). (72) Inventor: BLATZ, Philip, Strubing ; Box 3594, Greenville, DE 19807 (US). (74) Agents: KAEDING, Konrad, S. et al.; E.I. du Pont de Nemours and Company, Legal/Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).		(81) Designated States: BR, CS, JP, KR, RU, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE). Published <i>With international search report.</i>
(54) Title: RECYCLING OF POLYVINYLBUTYRAL (57) Abstract Polyvinylbutyral recovered from its utility as a layer adhered to glass has value as a molding resin when combined with incompatible polymer such as polyolefin, having a melt temperature less than the decomposition temperature of the polyvinylbutyral and an anhydride-modified polymer providing compatibilization to the blend, with the resultant blend being capable of providing tough molded articles.		

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ES	Spain				

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TITLE

RECYCLING OF POLYVINYLBUTYRAL

FIELD OF THE INVENTION

This invention relates to the utilization of
5 waste polyvinylbutyral by recycling.

BACKGROUND OF THE INVENTION

Polyvinylbutyral (PVB) has achieved notable
and long standing success as the interlayer between two
sheets of glass to form for example the windshields of
10 automobiles.

When the PVB is recovered from its interlayer
application, e.g., from the manufacture of reject
windshields or from the "junking" of automobiles, it
would be the practice to dispose of the recovered PVB
15 by incineration. The same is true for PVB waste
recovered from the manufacture of PVB, e.g. as roll
trim, start up PVB sheeting, and defective PVB
sheeting.

SUMMARY OF THE INVENTION

20 The present invention finds a use for
recovered polyvinylbutyral that has value, instead of
the past use in waste disposal, more specifically a use
involving melt fabrication of the recovered
polyvinylbutyral into useful articles of commerce.
25 Thus, one embodiment of the present invention comprises
melt blending waste polyvinylbutyral with an
incompatible polymer having a melt processing
temperature less than the decomposition temperature of
the polyvinylbutyral and an effective amount of
30 anhydride-modified polymer to compatibilize the
resultant blend.

The incompatible polymer component can also
be a recycle resin so the present invention
contemplates bringing together two "waste" resins to
35 produce a valuable result.

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The resultant melt blend can be used to mold articles by conventional means, e.g., injection molding or extrusion, to produce articles having high toughness and thereby widespread utility. The recovered PVB is permitted to impart toughness to the incompatible polymer by virtue of the presence of the compatibilizing polymer.

Another embodiment of the present invention is the composition resulting from the melt blending.

10 DETAILED DESCRIPTION OF THE INVENTION

The polyvinylbutyral (PVB) component of the melt blend can include additives which are often present in the resin to enable it to serve the interlayer utility between two sheets of glass, e.g., as automotive windshields and for architectural glass purposes. Such additives include primarily 20 to 30 weight percent plasticizer and about 0.1 to 0.5 weight percent of light stabilizer, based on total weight (PVB plus additives). Examples of plasticizers include tetraethylene glycol di-n-heptanoate, dihexyl adipate, dibutyl benzyl phthalate, triethylene glycol di-n-butylate, and diethylene glycol di-2-ethyl butyrate. Examples of light stabilizers include the benzotriazole and hindered amine light stabilizing compounds. The PVB itself will generally have a degree of polymerization of about 380 to 800 and a molecular weight of about 90,000 to 105,000 (weight average) and about 50,000 to 60,000 (number average).

The incompatible polymer component is preferably also a waste polymer, i.e. polymer waste resulting from the fabrication of useful articles or from the consumer use and discarding of such articles. Whether such polymer is virgin or recycle polymer, its incompatibility with PVB is manifested by blends with PVB having no appreciable improvement in physical properties and perhaps producing a deterioration in

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toughness. Examples of such polymers, which by themselves are not compatible with PVB include polyolefins, acrylonitrile/butadiene/styrene polymer (ABS), polystyrene, and a high impact polystyrene (HIPS). Examples of polyolefins include ethylene or propylene homopolymer or copolymer with each other and/or with other ethylenically unsaturated monomers. Preferably, the polyolefin is high density (about 0.945 to 0.960 g/cm³) polyethylene (HDPE) because this resin is available from recycling of post consumer rigid containers, e.g., from the base cups of polyethylene terephthalate (PET) soda bottles. Recycle HDPE, especially from base cups will have impurities associated with it, notably polypropylene from soda bottle labels, caps and cap retaining rings. Nevertheless, this HDPE is useful in the present invention. Another example of polyolefin is linear low density polyethylene (LLDPE) or low density polyethylene (LDPE), having a density of 0.920 to 0.945 and 0.915 to 0.930 g/cm³, respectively, which polymers may also be available from recycling. Preferably, the melt index of the polyolefin is greater than 5 g/10 min. All melt indices disclosed herein are measured at 190°C using a 2.160 Kg weight.

Recovered waste PVB by itself is not useful for interlayer utility any longer because the PVB contains contaminants or is otherwise out of specification and is therefore not acceptable as a glass interlayer product. The recovered PVB is melt fabricable, but because of its rubbery, low strength nature, it would have limited utility, hence the disposal by incineration or secure landfill instead of recycling of the PVB.

The incompatible polymers used in blends of the present invention are melt processible, even after

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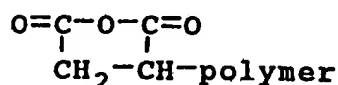
originating from waste. By melt processible is meant that the incompatible polymer softens sufficiently or melts under the application of heat and pressure, such as is obtained from an extrusion operation, to be intimately blended with other materials without any appreciable air pockets being present and can be shaped (melt fabricated) as desired. The incompatible polymers must, however, be melt processible at a temperature at which the PVB does not decompose. The decomposition temperature will vary somewhat, dependent on the time of exposure, which in turn will depend on the particular melt fabrication equipment used. Generally these incompatible polymers will be melt processible at temperature less than about 260°C and preferably less than 250°C. The toughness of these recycled polymers and even virgin polymer can be rather low, e.g. HDPE molded articles from low molecular weight HDPE such as used to make soda bottle base cups have a notched Izod impact strength of less than 2 ft-lb/in (107 J/m). HIPS has a toughness characterized by up to 7 ft-lb/in (374 J/m) notched Izod impact strength, depending on how much rubber is present, and this too can be improved by the process of the present invention. All notched Izod impact strengths reported herein refer to the procedure of ASTM D-256, with the impact testing being carried out at room temperature.

The combination of recovered PVB and recycled incompatible polymer after melt blending exhibits a notched Izod impact strength generally less than 1 ft-lb/in (53 J/m) for articles molded therefrom. This low toughness is indicative of incompatibility between these resins.

The third component of the melt blend in accordance with the present invention is a compatibilizing polymer. One embodiment of the

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anhydride modification of the polymer can be represented by the structure



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wherein the group attached to the polymer is succinic anhydride, which is typically obtained by reacting maleic anhydride (MANH) with the polymer such as described in U.S. patents 3,884,882 and 4,026,967. It has been found that anhydride groups on the polymer provide the compatibilizing effect as indicated by much higher toughness of articles molded from the three-component melt blend, e.g., a notched Izod impact strength which is typically at least 4x the notched Izod impact strength when the compatibilizing polymer is not present. Preferably, the notched Izod impact strength of blends of the present invention will be at least about 4 ft-lb/in (214 J/m), and more preferably at least 8 ft-lb/in (428 J/m). Recycled HDPE base cup has a notched Izod impact strength of only about 0.5 ft-lb/in (26.7 J/m), and this resin can be toughened to at least about 2.5 ft-lb/in (133 J/m) by melt blended with PVB (40 weight percent) and 10% compatibilizing polymer. Generally, the anhydride groups of the compatibilizing polymer are obtained by grafting onto the polymer by conventional means using such carboxylic acids or anhydrides as maleic anhydride or nadic anhydride. The anhydride groups will generally represent about 0.5 to 2.5 weight percent, and more often, about 0.75 to 2.0 weight percent, based on the total weight of the compatibilizing polymer.

Generally, the polymer portion of the compatibilizing polymer will have at least some affinity for the incompatible polymer as represented by the compatibilizing polymer being fully dispersible in

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molten incompatible polymer. The compatibilizing polymer will often be derived from ethylene monomer or comonomer as a principal constituent. Examples of compatibilizing polymers include high density

5 polyethylene grafted with about 1% maleic anhydride (MANH), linear low density polyethylene grafted with maleic anhydride, polypropylene grafted with maleic anhydride, ethylene/propylene/diene terpolymer grafted with about 1.5 to 1.75 weight percent MANH, ethylene/n-

10 butyl acrylate/carbon monoxide terpolymer grafted with about 1 weight percent MANH, ethylene/vinyl acetate/carbon monoxide terpolymer grafted with about 1 weight percent MANH, ethylene/propylene copolymer grafted with about 1 weight percent MANH, ethylene/n-

15 butyl acrylate copolymer grafted with about 1 weight percent MANH, ethylene/ethyl acrylate copolymer grafted with about 1 weight percent MANH, ethylene/vinyl acetate copolymer grafted with about 1 weight percent MANH, and styrene-ethylene/butene-styrene block

20 copolymer grafted with about 2 weight percent MANH. The MANH in these polymers means that the grafting agent is maleic anhydride and the result of anhydride modification of the polymer is the presence of succinic anhydride groups pendant from the polymer. The weight

25 percent of MANH refers to the proportion of succinic anhydride present on the polymer. Examples of nadic anhydride modified polymers useful as the compatibilizing polymer component include nadic anhydride-modified polyethylene and nadic anhydride-

30 modified ethylene/propylene/diene terpolymer. The selection of compatibilizing polymer will depend on the particular incompatible polymer present in the blend, i.e. the compatibilizing polymer should have affinity for the incompatible polymer. The anhydride

35 functionality of the compatibilizing polymer provides the compatibility with the PVB component.

The PVB component is collected from waste resulting from PVB manufacture and from the manufacture of glass laminates wherein the PVB in sheet form is needed as an interlayer between sheets of glass. This component may also be recovered from the glass laminates after their manufacture and from "junked" automobiles. The resultant waste resin can be prepared for utilization in accordance with the present invention by comminution into particles which can be blended with the other components of the blend.

The incompatible polymer component can be virgin resin, but if it is HDPE recycle resin, it can be obtained from the granulation of HDPE containers or from the base cups of PET bottles. These base cups can be removed from the PET bottles with sufficient force to overcome the strength of the glue bonding the base cup to the bottle, or the entire bottle, including base cup, can be subjected to granulation into flake form, hot caustic soak (3% NaOH aqueous solution at 88°C) to detach the glue, thereby freeing up the HDPE flakes from the PET flakes, followed by separation of the lighter HDPE flakes (specific gravity of about 0.94 - 0.96) from the heavier PET flakes (specific gravity of about 1.4). The recycle HDPE may contain a minor proportion, e.g., up to 20 weight percent of polypropylene, by virtue of the association of polypropylene with HDPE in container applications as the base cups, caps, retaining rings, and labels therefor.

The PVB, incompatible polymer, and anhydride modified (compatibilizing) polymer can be melt blended in apparatus which thoroughly disperses these resins within one another in the molten state. The proportion of PVB and incompatible polymer can depend on the relative availability of these polymers, but generally

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about 20 to 60 weight percent PVB, 20 to 60 weight percent incompatible polymer, and about 2.5 to 20 weight percent of the compatibilizing polymer will be present, based on the weight of the blend (combined weight of the three components) will be present. This amount of compatibilizing polymer in the blend will generally be effective to substantially improve the toughness of the blend over the blend without the compatibilizing polymer. The effectiveness of the compatibilizing polymer will vary for a given amount as the amount of anhydride functionality is varied. For example, an increase in presence of anhydride groups will generally increase effectiveness. The amount of compatibilizer polymer used and the amount of anhydride groups present therein will also be influenced by economical considerations as well as the toughness results needed for particular applications. The preferred proportion of the compatibilizing polymer is about 5 to 15 weight percent based on the weight of the blend. Preferably the melt blending accomplishes a fine dispersion of the PVB within the matrix of incompatible polymer, in which condition the compatibilizing polymer enables the PVB to toughen the incompatible polymer to provide a tough blend. From these compositions, it will be seen that articles molded from the blends can consist of at least about 80 weight percent of waste resin. The melt blend can be formed into molding granules for subsequent melt processing, i.e., melt fabrication in articles for consumer use.

Numerous articles having widespread household and industrial utility can be melt fabricated from blends of the present invention. For example, blends of the present invention can be used to mold trash cans and lids and drain pans for oil from automobiles that receive and withstand rough handling.

The molding of articles from the blends of the present invention can be by conventional melt fabrication processes such as injection molding and extrusion.

5 Examples of the present invention are presented hereinafter; parts and percents are by weight unless otherwise indicated.

EXAMPLES

EXAMPLE 1

10 A mixture of 27.5 g (50%) flakes of HDPE recycle resin from soda bottle base cups, 22.0 g (40%) recovered PVB, and 5.5 g (10%) of ethylene (71%)/propylene (23%) /1.4-hexadiene (6%) copolymer (weight average molecular weight of 230,000) grafted
15 with 1.8% succinic anhydride groups (wherein the chemical groups grafted to the copolymer are succinic anhydride) was melt blended at 190°C for five minutes using a laboratory batch mixer which formed molding granules from the blend. The melt index of the HDPE in
20 the base cup is about 30 g/10 min., but the presence of the polypropylene contaminant therein reduces the melt index to about 6.5 g/10 min. Flex bars (1/8 in, 3.2 mm) and tensile bars (1/8 in, 3.2 mm) were compression molded from the molding granules at 190°C and these
25 test bars were tested for notched Izod impact strength and tensile elongation to give the following results: notched Izod impact strength of 14.7 ft-lb/in (785 J/m) and elongation of 290%.

By way of comparison, when the compatibilizer
30 polymer was omitted from the blend and the composition of the blend was 60/40 of HDPE and PVB, the notched Izod impact strength was only 0.4 ft-lb/in (21 J/m) and the elongation was only 74%. When both the compatibilizer copolymer and the PVB were omitted, the

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notched Izod of the recycled HDPE base cup was only 0.5 ft-lb/in (26.7 J/m).

By way of further comparison, when the compatibilizing polymer contained only 0.5 weight percent succinic anhydride groups, the notched Izod impact strength was less than 4 ft-lb/in (214 J/m). An increased proportion of polymer would be expected to provide further improvement.

By way of further comparison, when the amount of compatibilizer polymer was reduced to 7.5% and then 5%, with the HDPE component being correspondingly increased to 52.5% and 55% respectively, the notched Izod impact strengths were 14.7 ft-lb/in (785 J/m) and 14.0 ft-lb/in (748 J/m), respectfully.

When the copolymer (third component) had no succinic anhydride groups, the compatibilizing effect was much less. For example, 10% of ethylene (67.5%)/n-butyl acrylate (23.5%)/methacrylic acid (9%) in the 50 HDPE/40 PVB blend gave a notched Izod impact strength of 7.1 ft-lb/in (379 J/m) for molded test bars from the blend, indicating the greater effectiveness of the anhydride-containing polymer in compatibilizing the blend. In another example, 10% of ethylene (76.5%)/n-butyl acrylate (23.5%)/methacrylic acid (9%) which was 70% neutralized with Zn ions to form ionomer gave a notched Izod impact strength of only 5.3 ft-lb/in (282 J/m) for bars molded from the HDPE (50%)/PVB (40%) blend. When the third component was 10% ethylene (80%)/isobutyl acrylate (10%)/methacrylic acid (10%), the notched Izod impact strength was less than 4 ft-lb/in (214 J/m). When the ethylene copolymer was neutralized with sodium ions rather than zinc ions, test bars molded from resultant HDPE (50%)/PVB (40%) blend also gave an notched Izod impact strength of less than 4 ft-lb/in (214 J/m).

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By way of further example of third components that did not work as a compatibilizer, ethylene/n-butyl acrylate/glycidyl methacrylate copolymer, and ethylene/n-butyl acrylate/carbon monoxide, which are known tougheners for polyester resin, had little effect as compatibilizer resin in the present invention. In experiments in which these resins were separately substituted for the 10% succinic anhydride modified copolymer described at the beginning of this Example, test bars of the resultant HDPE (50%)/PVB (40%) blends gave notched Izod impact strengths of less than 4 ft-lb/in (214 J/m).

Example 2

The procedure of this Example was essentially the same as set forth in the first paragraph of Example 1, except that the compatibilizer polymer was 10% styrene-ethylene/butene-styrene block copolymer grafted with 2% succinic anhydride available as Kraton® FG 1901X, and the notched Izod impact strength of test bars molded from the HDPE (50%)/PVB (40%) blend was 14.6 ft-lb/in (780 J/m).

Example 3

The procedure of this Example was essentially the same as in the first paragraph of Example 1 except that the proportion of HDPE and PVB was varied in the blend, with the 10% of the compatibilizer polymer component being held constant, to give the following notched Izod impact strength for test bars molded from the blends.

30

<u>HDPE</u>	<u>PVB</u>	<u>IMPACT STRENGTH</u>	
<u>%</u>	<u>%</u>	<u>ft-lb/in</u>	<u>J/m</u>
60	30	8.7	465
50	40	10.4	555
45	45	10.7	571
30	60	11.2	598

Example 4

The procedure of the first paragraph of Example 1 was essentially repeated except that (a) the amount of compatibilizer polymer was only 5% and the amount of HDPE was 55% and (b) the blending of the three components was carried out on a twin-screw compounding extruder. Test bars molded from the resultant blend exhibited a notched Izod impact strength of 10 ft-lb/in (534 J/m) and an elongation of 230%.

Example 5

The procedure of the first paragraph of Example 1 was essentially repeated except that the composition that was melt blended consisted of 48% of the recycled HDPE base cup, 40% recovered PVB, 10% of the E/P/diene copolymer and 2% carbon black concentrate (50:50 LDPE and carbon black) and the blending of these components was carried out in a twin screw compounding extruder operating to produce a melt temperature of about 224°C and an extrusion rate of only 13 lb/hr (5.9 kg/hr) so as to give sufficient residence time of the melt blend within the extruder to finely disperse the PVB within the HDPE and to permit the interaction between the copolymer and the PVB to obtain a compatibilized blend having a melt index of 1.75 g/10 min. The molding granules from the extruder were injection molded into flex bars and tensile bars which exhibited the following properties: flex modulus = 21.5 Kpsi (148 MPa), notched Izod impact strength of 11 ft-lb/in (586 J/m), tensile strength of 2050 psi (14.1 MPa) and elongation of 335%.

As many widely different embodiments of this invention may be made without departing from the scope and spirit thereof, it is to be understood that this invention is not limited to the specific

embodiments thereof except as defined in the appended claims.

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CLAIMS

1. A process for recovering value from waste polyvinylbutyral, comprising melt blending said polyvinylbutyral with incompatible polymer having a melt processing temperature less than the decomposition temperature of said polyvinylbutyral and an effective amount of anhydride-modified polymer to compatibilize the resultant blend.
2. The process of Claim 1 wherein the proportion of polyvinylbutyral is about 20 to 60 weight percent, the proportion of incompatible polymer is about 20 to 60 weight percent, and the proportion of anhydride-modified polymer is about 2.5 to 20 weight percent, based on the combined weight of these components.
3. The process of Claim 1 wherein the compatibilization of the resultant blend is indicated by articles from it having a notched Izod impact strength of at least 4 ft-lb/in (214 J/m).
4. The process of Claim 1 wherein the polyvinylbutyral being recovered is derived from the manufacture of polyvinylbutyral, the manufacture of glass laminates wherein the polyvinylbutyral is an interlayer between glass sheets of the laminates, or from said glass laminates after such manufacture.
5. The process of Claim 1 wherein the anhydride-modified polymer is succinic anhydride-modified ethylene copolymer.
6. The process of Claim 1 wherein said melt processing includes melt fabricating an article from said blend.
7. Melt blended composition comprising polyvinylbutyral, incompatible polymer having a melt temperature less than the decomposition temperature of said polyvinylbutyral, and an effective amount of anhydride-modified polymer to compatibilize the blend.

8. The melt blended composition of Claim 7
wherein said polyvinylbutyral and incompatible polymer
are both waste polymer.

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/US 92/06000

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 C08L51/06; C08J11/06; C08L29/14		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	C08L ; C08J	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
P,X	US,A,5 130 370 (MONSANTO) 14 July 1992 see the whole document	1,2,4
A	EP,A,0 167 455 (KAWASAKI STEEL) 8 January 1986 see claims 1,6-9; example 3	1-5
P,A	EP,A,0 471 658 (MONSANTO) 19 February 1992 see page 2, line 12 - line 17; claim 1	1,4
<p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"A" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
26 OCTOBER 1992	- 2. 11. 92	
International Searching Authority	Signature of Authorized Officer	
EUR PEAN PATENT OFFICE	ENGEL S.L. <i>H. Engel</i>	

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

US 9206000
SA 62629

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
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